

PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

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| Applicant's or agent's file reference 154874/LET/BF | FOR FURTHER ACTION See Form PCT/IPEA/416 | |
| International application No. PCT/NO 2003/000263 | International filing date (day/month/year) 30.07.2003 | Priority date (day/month/year) 31.07.2002 |
| International Patent Classification (IPC) or national classification and IPC C01B33/03, C01B33/029, B01J12/02, B01D49/00, B01D45/06 | | |
| Applicant Sörlandets Teknologisenter AS et al | | |

- This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
- This REPORT consists of a total of 4 sheets, including this cover sheet.
- This report is also accompanied by ANNEXES, comprising:
 - ☒ (sent to the applicant and to the International Bureau) a total of 3 sheets, as follows:
 - ☐ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
 - ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
 - ☐ (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) _____, containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).
- This report contains indications relating to the following items:

| | | |
|-------------------------------------|--------------|---|
| <input checked="" type="checkbox"/> | Box No. I | Basis of the report |
| <input type="checkbox"/> | Box No. II | Priority |
| <input type="checkbox"/> | Box No. III | Non-establishment of opinion with regard to novelty, inventive step and industrial applicability |
| <input type="checkbox"/> | Box No. IV | Lack of unity of invention |
| <input checked="" type="checkbox"/> | Box No. V | Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement |
| <input type="checkbox"/> | Box No. VI | Certain documents cited |
| <input type="checkbox"/> | Box No. VII | Certain defects in the international application |
| <input type="checkbox"/> | Box No. VIII | Certain observations on the international application |

| | |
|---|---|
| Date of submission of the demand 25.02.2004 | Date of completion of this report 15.10.2004 |
| Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. +46 8 667 72 88 | Authorized officer Mårten Hulthén/ELY Telephone No. +46 8 782 25 00 |

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/NO 2003/000263

Box No. I Basis of the report

1. With regard to the language, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.

- ☐ This report is based on a translation from the original language into the following language _____, which is the language of a translation furnished for the purposes of:
- ☐ international search (under Rules 12.3 and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4)
 - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)

2. With regard to the elements of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

- ☐ the international application as originally filed/furnished
- ☒ the description:
- pages 1 - 13 _____ as originally filed/furnished
- pages* _____ received by this Authority on _____
- pages* _____ received by this Authority on _____

- ☒ the claims:
- pages _____ as originally filed/furnished
- pages* _____ as amended (together with any statement) under Article 19
- pages* 11 - 13 received by this Authority on 26.08.2004
- pages* _____ received by this Authority on _____

- ☒ the drawings:
- pages 1 - 6 _____ as originally filed/furnished
- pages* _____ received by this Authority on _____
- pages* _____ received by this Authority on _____

- ☐ a sequence listing and/or any related table(s) – see Supplemental Box Relating to Sequence Listing.

3. ☒ The amendments have resulted in the cancellation of:

- ☐ the description, pages _____
- ☒ the claims, Nos. 8 - 16
- ☐ the drawings, sheets/figs _____
- ☐ the sequence listing (*specify*): _____
- ☐ any table(s) related to the sequence listing (*specify*): _____

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

- ☐ the description, pages _____
- ☐ the claims, Nos. _____
- ☐ the drawings, sheets/figs _____
- ☐ the sequence listing (*specify*): _____
- ☐ any table(s) related to the sequence listing (*specify*): _____

* If item 4 applies, some or all of those sheets may be marked "superseded."

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/NO 2003/000263

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | | |
|-------------------------------|--------|------------|-----|
| Novelty (N) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |
| Inventive step (IS) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |
| Industrial applicability (IA) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |

2. Citations and explanations (Rule 70.7)

Documents considered being of particular relevance:

D1 US 4668493

D2 US 4272488

The invention relates to the production of pure silicon by the reduction of a silicon precursor with hydrogen in a reactor. The silicon precursor is introduced into the reactor through a tube, which is arranged coaxially with an outer tube trough which hydrogen is fed.

D1 discloses a similar process as the invention for producing silicon but does not disclose a coaxial feed system for introducing a silicon precursor and hydrogen. Therefore, D1 has been reconsidered not to be of particular relevance.

D2 (abstract) reveals a reactor for producing silicon from silane and hydrogen that comprises an "elongated container" with an "inert lining" and is equipped with an "injection system". It also involves a coaxial feed system for hydrogen and silane.

The invention differs from D2 in that hydrogen gas functions as a cooling medium for the introduced silicon precursor and that a steep temperature gradient is held within the reactor as stated in claims 1 and 7. Further, the reactor is equipped with a constant level outlet system as stated in claims 5 and 7. Consequently, the invention as defined in independent claims 1, 5 and 7 is novel.

The stated differences imply improvements in avoiding premature decomposition of the silicon precursor and the fouling of feed lines when producing high grade silicon.

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Supplemental Box

In case the space in any of the preceding boxes is not sufficient.

Continuation of: BOX V

Therefore, the invention as defined by claims 1-7 is considered to involve an inventive step. The invention is also considered to fulfil the criteria of industrial applicability.

Claims

1. A method for producing Solar Grade Silicon by thermal decomposition of a silicon precursor in the presence of an excess of hydrogen by separately introducing hydrogen gas and the silicon precursor in a vertically oriented reaction chamber in which the lower portion holds a pool of molten silicon, the surface of which the reactants are directed toward and react during the formation of essentially elementary silicon and hydrogen chloride, off gases and by-products formed are withdrawn from the upper portion of the column; silicon formed is withdrawn from the lower portion of the reactor, characterized in that the fluid silicon precursor is introduced at ambient temperatures through a pipe arranged coaxially in a feed pipe for hydrogen gas, where the hydrogen gas functions as a cooling medium for the introduced silicon precursor, in that a steep temperature gradient is held within the reactor, and that the reactants are conducted at great speed toward the surface of the molten silicon by rapid expansion of the silicon precursor and hydrogen gas, and brought to react in the lower warm portion of the reactor, in that any formed silicon chloride reacts with the excess hydrogen in the upper portion of the reactor and falls as particulate solid silicon.
2. The method according to Claim 1 characterized in that the silicon precursor is trichlorosilane, silane, tetrachlorosilane, or other halogenated silanes, preferably trichlorosilane.
3. The method according to Claim 1 or 2, characterized in that the lower portion of the reactor is held at a temperature above the

melting point of silicon, 1410 °C, and the cold upper portion is held at a temperature under 400 °C.

4. The method according to Claim 1, 2 or 3,
c h a r a c t e r i z e d i n that the level of
molten silicon in the reactor is held constant by a
constant level system for continuous removal.
5. A reactor for the practice of the method according to
the preceding Claims comprising an elongated
container (1) with an inert lining, where the
container is equipped with an injection system (2)
for a silicon precursor, preferably trichlorosilane,
and hydrogen, and an outlet (5) for unconverted
hydrogen HCl and any by-products,
c h a r a c t e r i z e d b y a constant level
outlet system (6) for produced, liquid silicon, that
the feed system (2) comprises an outer pipe (4) for
introducing hydrogen gas and a pipe (3) arranged
coaxially with pipe(4) for the introduction of fluid
silicon precursor, and where the height of the
reactor is sufficient to establish the necessary
steep temperature gradient.
6. Reactor according to Claim 5,
c h a r a c t e r i z e d i n that the temperature
in the lower portion of the reactor is held at a
temperature above the melting point of silicon, 1410
°C, and the cold upper portion is held at a
temperature under 400 °C.
7. Use of the method according to Claims 1-4 and the
reactor according to Claims 5-6 for the production of
Solar Grade Silicon in an integrated, essentially
closed process by reacting low grade silicon with
hydrogen chloride gas during the formation of
trichlorosilane which is introduced into a reactor
according to Claim 5, after which unconverted

hydrogen and hydrogen chloride are separated and hydrogen chloride gas is used for the conversion of additional low grade silicon and the hydrogen gas is introduced as a reactant in the process.

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